FORMULATION OF A NEW LIQUID FLUX FOR HIGH TEMPERATURE SOLDERING

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ABSTRACT

Through-hole soldering is alive and well in the electronics industry, despite its predicted demise at the hands of surface mount technology. Through-hole soldering is largely used for connectors, switches, and other components that require a high solder joint strength. High reliability circuit assemblies will likely use through-hole technology for many years to come.

Thermally demanding circuit boards can be quite difficult to solder due to the high temperatures and long contact times required. Many liquid fluxes available on the market cannot withstand these type of soldering conditions resulting in poor hole fill, bridging and other defects. The goal of this project was to formulate a new liquid flux designed to give optimal soldering performance with thermally demanding circuit boards.

The development of this new liquid flux was guided by these key characteristics.

1. Withstand soldering temperatures up to 290 °C with long contact times.
2. Produce optimal hole fill and minimize bridging or other defects.
3. Residue must be easy to wash using de-ionized water, and produce very little foam.
4. Flux must be completely halide and halogen free.
5. Able to be used in leaded and lead-free wave and selective solder systems.

This paper presents the process of developing this new liquid flux. Laboratory and beta-site test results for existing fluxes are compared to this new flux. The result is a unique product that can help solve the challenges of through-hole soldering thermally demanding assemblies.

Key words: formulation, liquid flux, wave solder, selective solder, high temperature, soldering, halide free

INTRODUCTION

Through hole soldering is used to ensure high solder joint strength [1]. Certain types of connectors are designed to be used with repetition. This repetitive usage can weaken the solder joint over time. Through hole solder joints provide the strength needed for this type of application.

Historically, through hole soldering has been done with a wave solder process. This process involves the following steps (Figure 1).

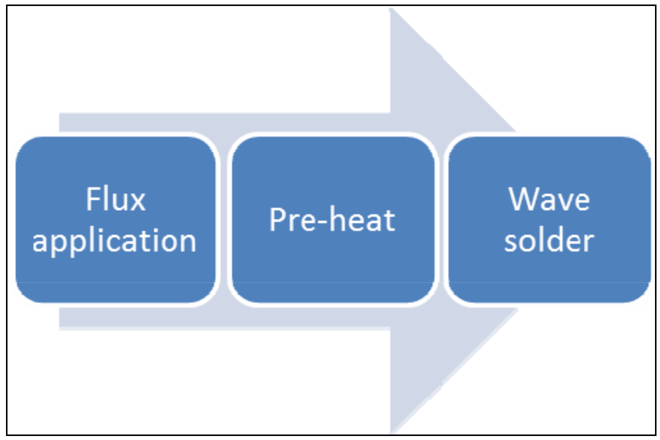

Figure 1: Wave soldering process

Flux is applied through a foaming or spray flux system. In both cases the flux is applied from the bottom of the circuit board and must flow up through the holes. The amount of flux applied is typically measured by weight [2]. Flux manufacturers recommend a range of flux mass per unit area of the circuit board. The weight of the flux applied directly relates to the amount of active material that is available to aid in soldering. Pre-heating helps to increase the temperature of the circuit board closer to soldering temperature. Pre-heating also removes some of the solvents of the flux. This is especially important for VOC free fluxes which typically use water as the primary solvent. The wave soldering step solders all of the holes nearly simultaneously as the board passes over the wave.

In recent years, selective soldering has become more popular. The selective soldering process is similar to the wave soldering process but has notable differences (Figure 2).

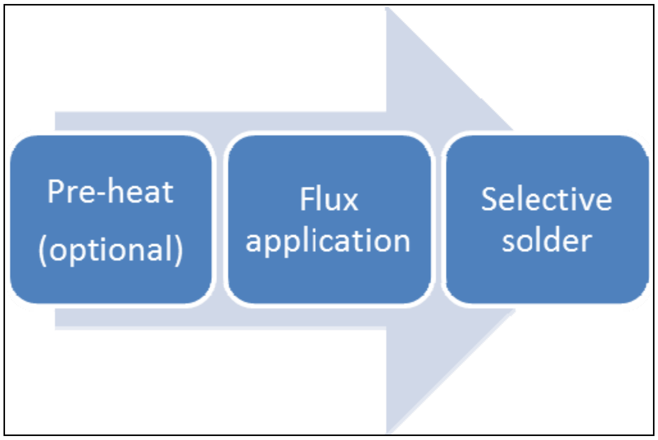

Figure 2: Selective soldering process

The selective soldering process solders one location on the board at a time. Pre-heating is an option in selective soldering. Pre-heating can be done before or during soldering. Systems that do not use pre-heating utilize the solder itself to both heat and solder the holes. Some selective soldering systems use heated nitrogen gas to provide heat to the holes. Flux is applied to a location, then selective soldering occurs. This is repeated until all locations have been soldered. The solder temperature and contact time depend largely upon the board design. In general, selective solder temperatures are 30 to 50 °C higher than wave solder temperatures.

In selective soldering, flux application is done using spray or "drop-jet" type systems. Flux is applied from the bottom side of the board, and the flux must wet the holes to the top side of the board. Spray systems will typically overspray around the hole area applying excess flux to the bottom side of the board. This excess flux will not be heated by the solder and therefore remains chemically active. This causes concern about the long term reliability of circuit boards soldered with no clean fluxes. Water soluble fluxes cause less concern because washing is required after soldering. Remaining active flux materials will be removed by the washing process.

Some circuit boards are made with high layer counts, high copper weights, and metal cladding. The intention of these designs is to provide heat sinking to draw heat away from sensitive components. Unfortunately these type of circuit boards are difficult to solder. The hole barrels must be heated to the melting point of the solder in order for solder to wet to the top of the barrel. Long and higher temperature pre-heat settings are required. Lead-free solder temperatures in excess of 290 °C for wave soldering and 315 °C for selective soldering, and long contact times are used. Fluxes used for thermally demanding circuit boards must be able to tolerate these conditions and still provide excellent wetting. The flux residues must be easy to wash after high temperature exposure.

New water soluble fluxes should be formulated in order to meet the needs of both wave and selective soldering systems. These fluxes have to be able to be applied by all types of fluxing systems. The fluxes cannot cause corrosion or other chemical attack in areas of the circuit board that are not heated. The residues both heated and un-heated must be easy to wash off. New flux formulations have to be useable for standard circuit board designs and thermally demanding assemblies.

EXPERIMENTAL METHODOLOGY

Formulation of liquid fluxes is done using part creativity, part prior knowledge, and part trial and error. It is difficult to go into detail about the process of formulation without disclosing trade secrets. The basic process is listed below.

1. Make small batches of test fluxes.
2. Test the fluxes in the laboratory using test coupons and a lab solder pot.
3. Repeat steps 1 and 2 until the desirable properties of the flux are achieved.
4. Make a larger batch of a test flux and send to beta sites for testing using wave and selective solder systems.
5. Using feedback from the beta sites refine the flux formulation.
6. Finalize the flux formulation.

IPC standard methods [3] for solderability were used in lab testing. Standard immersion fluxing and solder float testing were used. The circuit boards were evaluated using criteria that is more rigorous than industry standard criteria [4]. J-STD-001F states that 75% of the barrel must be vertically filled for Class 2 and 3 product. Hole fill was measured by counting only the holes that were 100% vertically filled. Then the percentage of filled holes out of the total possible was calculated.

Standard Solderability Test Board

Initial testing was done using this solderability circuit board (Figure 3).

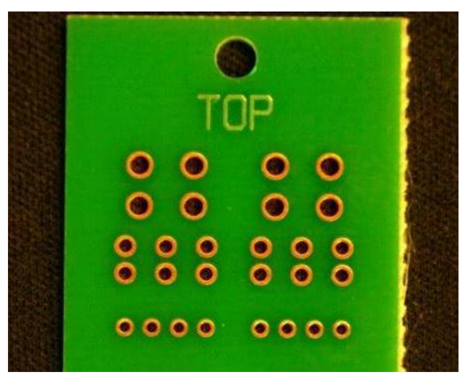

Figure 3: Standard solderability test board

This circuit board is 0.062 inches thick (1.57 mm). This circuit board is double sided and is made using 0.5 ounce copper weights. It has three hole sizes: 0.055 inch diameter (1.40 mm), 0.039 inch diameter (0.99 mm), and 0.032 inch diameter (0.81 mm). The circuit board does not have a solderable finish applied. Soldering was done to bare copper. This circuit board is used for laboratory solderability testing, and is representative of relatively easy to solder circuit boards.

Thermally Demanding Test Board

A new test board was designed to be thermally demanding (Figure 4) and more challenging for hole fill testing.

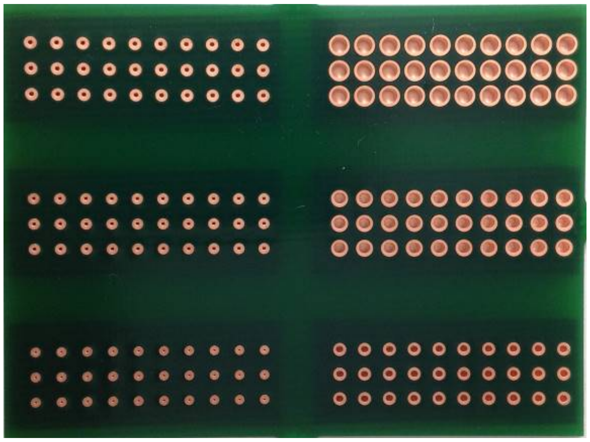

Figure 4: Thermally demanding solderability test board

This thermally demanding test board is 0.092 inches (2.34 mm) thick. It is a 4 layer board using 2 ounce copper weights on each layer. There are six drilled hole sizes: 0.010 inch (0.25 mm) diameter, 0.0156 inch (0.40 mm) diameter, 0.020 inch (0.51 mm) diameter, 0.0295 inch (0.75 mm) diameter, 0.0453 inch (1.15 mm) diameter, and 0.061 inch (1.55 mm) diameter. The inner layers are copper ground planes. The holes are organized in arrays of 3 rows of 10 holes each (Figure 5).

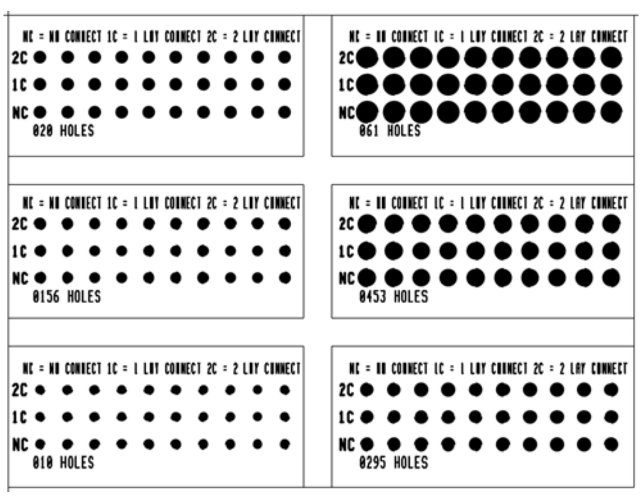

Figure 5: Design of thermally demanding solderability test board

One row of each hole size is not connected (NC) to the inner layer ground planes, and these holes require less heat to solder. One row is connected to one inner layer ground plane (1C), which makes these holes more thermally demanding. One row is connected to both inner layer ground planes (2C), which makes these holes the most thermally demanding. The circuit board does not have a solderable finish applied. Soldering was done to bare copper.

The finished hole sizes of the thermally demanding solderability test board were measured and aspect ratios calculated (Table 1).

Table 1: Hole sizes and aspect ratios of thermally demanding test board

Hole Size Drilled (mils)	Hole Size Finished (mils)	Aspect Ratio
10	7	13 : 1
15.6	13	7 : 1
20	16	5.8 : 1
29.5	27	3.4 : 1
45.3	43	2 : 1
61	60	1.5 : 1

The largest aspect ratios of 7:1 and 13:1 were incorporated into this test design in order to challenge the soldering ability of the fluxes.

Flux Formulations

The fluxes that were tested were all water soluble, neutral pH formulations. All of these formulations use isopropanol (IPA) as the main solvent. These formulations were named with letter codes and the properties of the fluxes are listed in Table 2 below.

Table 2: Properties of fluxes tested

Flux	Density (g/cc)	Halogens	Non-volatile content (% wt)
Current	0.87	yes	20
SP 20%	N/A	yes	20
W	0.92	yes	40
X	0.93	yes	40
Y	0.90	yes	37
Z	0.92	yes	38
A	0.91	no	26
B	0.91	no	27
C	0.90	no	27
D	0.88	no	28
E	0.85	no	24
F	0.85	no	26
G	0.85	no	26
I	0.86	no	25

The fluxes tested include a current formulation that has been sold for several years and a special product which was made by mixing a gel flux at 20% by weight into IPA. The gel flux did not completely dissolve into the IPA and formed a slurry, therefore an accurate density was not able to be obtained. The rest of the fluxes on this list are test formulations which vary in density, halogen content and non-volatile content. Initially halogens were included in the test formulations, but it was desirable to create a halogen free flux, so later formulations were all completely halide and halogen free. It is not practical to measure acid number of these fluxes due to the fact that they are neutral in pH. Acid number is also not a good measure of activity [5]. High acid number fluxes may not withstand high temperature soldering, while low acid number fluxes might perform well at high temperatures depending upon the ingredients used. The solids content gives some indication of the activity level of the fluxes. The true activity of these fluxes is shown through more practical solder testing and evaluation of hole fill.

The requirements for determination of solids content are given in section 3.4.2.1 of IPC J-STD-004 [6]. In this paragraph the terms solids and non-volatile are intended to refer to the amount of material that does not evaporate when test method 2.3.34 is used. This test method uses relatively low temperatures to determine the solids content of liquid fluxes (50 – 85 °C). These temperatures are high enough to drive off low boiling solvents like IPA (B.P. 82.5 °C), but not high enough to evaporate off most of the other ingredients. The non-volatile contents listed in Table 1 were determined using this IPC standard method.

Please note that the term "Non-volatile" content is used rather than "solids" content in Table 1. The term "solids" content leads one to believe that it represents the total amount of dissolved solid or crystalline materials in the flux. The non-volatile content of these liquid fluxes includes materials that are liquids at room temperature, but do not evaporate during testing. This distinction can be important for selective soldering applications. Higher solids content fluxes can cause clogging issues [7], especially with drop jet type fluxing systems. Clogging typically occurs due to crystallization of dissolved solid materials. Fluxes that are high in non-volatile content can be relatively low in dissolved solids content and will work well with drop jet fluxing systems.

RESULTS

Standard Solderability Test Board Results

Initial testing was done on all fluxes using the standard solderability test board (Figure 3). Circuit boards were fluxed, soldered and the percentage of holes filled was calculated for each hole diameter. The solder used was Sn/Cu/Ni/Ge which is sold under the trade name SN100C®. Testing was repeated for 3 solder pot temperatures: 260, 280, and 300 °C. Washing was done within a few minutes after soldering using de-ionized water with no agitation, and the boards were inspected for flux residues.

The hole fill results at 260 °C are sorted by flux formulation in Figure 6.

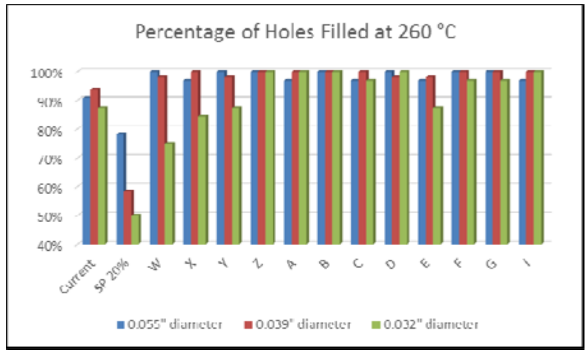

Figure 6: Hole fill results for the fluxes at 260 °C

Hole size has an effect on solderability. The percentage of holes filled decreases with decreasing hole size. This is especially noticeable for the SP20% flux and fluxes W, X, and Y. SP20% flux was a slurry which had trouble wetting the holes completely. This caused the soldering performance to be much worse than the other fluxes.

Water wash results were varied for the fluxes (Table 3). Some fluxes left faint residues over the solder mask, and others left waxy type residues mainly around the solder joints. Some fluxes washed clean and left no residues.

Table 3: Water wash results for the fluxes

Flux	Water Wash Results
Current	Washed clean. No residues
SP 20%	Washed clean. No residues
W	White haze over surface
X	White haze over surface
Y	White haze over surface
Z	Faint residue on surface, but improved over W, X, and Y
A	Gratuitous white waxy residue, especially around solder joints
B	Waxy residue
C	Waxy residue
D	Waxy residue
E	Washed clean. No residues
F	Washed clean. No residues
G	Washed clean. No residues
I	Washed clean. No residues

Pictures of a flux that left residues are compared to a flux that washed clean in Figure 7.

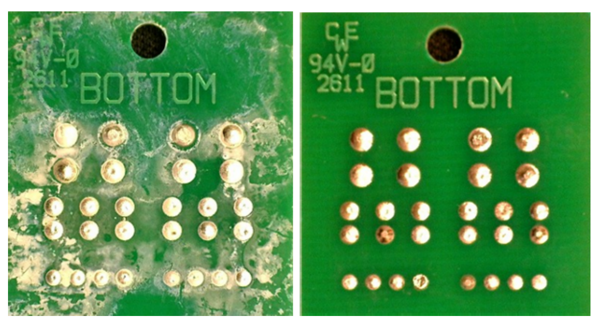

Figure 7: Flux residues remaining after washing (left) and a cleanly washed flux (right)

After the initial round of testing, the fluxes were evaluated for soldering performance and wash ability. Some of the fluxes were eliminated from testing, and the rest were run at higher temperatures. The results for testing at 280 °C are shown in Figure 8.

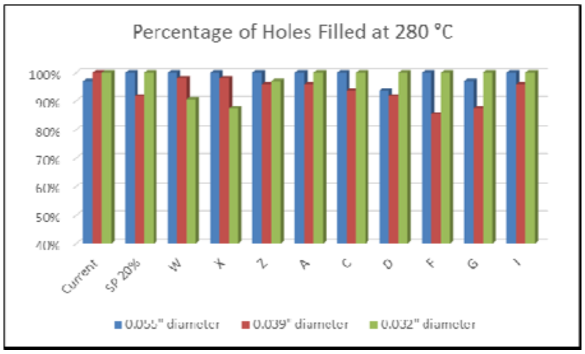

Figure 8: Hole fill results for the fluxes at 280 °C

The current flux and SP20% flux both performed better in hole fill testing at 280 °C than they did at 260 °C. The increased heat provided by the solder helped the flux and solder to flow to the top of the holes. In general, the 0.039" (0.99 mm) diameter holes showed lower hole fill than the other hole sizes. The larger and smaller holes gave nearly 100% hole fill percentages.

Again after this testing, some fluxes were eliminated from contention and the others were tested at 300 °C. The results for testing at 300 °C are shown in Figure 9.

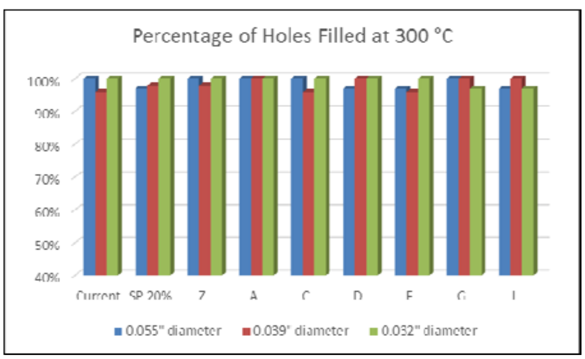

Figure 9: Hole fill results for the fluxes at 300 °C

Hole fill results were very similar for all of these fluxes at 300 °C. Most fluxes gave hole fill percentages near 100%. In general, hole fill performance improved with increasing solder temperature. Murphy [8] reported that temperature had a more dramatic effect on hole fill than flux amount. This mirrors our results with increasing solder temperature. The ideal flux needs to perform well at all of the temperatures tested.

Water wash performance was very similar at each temperature for each flux. Fluxes that left a residue at 260 °C left a very similar residue at 280 °C and 300 °C. As testing progressed, fluxes that left residues were eliminated from testing. The fluxes that washed cleanly at 260 °C also washed cleanly at 280 °C and 300 °C (Figure 10).

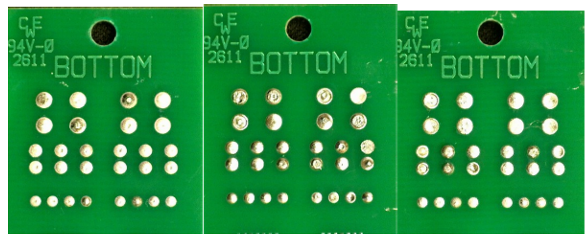

Figure 10: Optimal flux wash performance at 260 °C (left), 280 °C (center), and 300 °C (right)

The standard test board design helped to differentiate between these fluxes in early testing. As testing progressed, the fluxes seemed to perform very similarly on the standard test board design. This created the need for a more challenging test vehicle.

Thermally Demanding Test Board Results

Select fluxes were tested with the thermally demanding solderability test board (Figure 4). The same basic process was used for these test boards and for the standard solderability test boards. The fluxes tested include the current flux, SP 20%, formula Z, and formula I. The results for solderability testing at 260 °C are shown in Figures 11 through 14. The blue bars represent the holes that are connected to both inner layer ground planes (2C). The red bars represent the holes that are connected to only 1 inner layer ground plane (1C). The yellow bars represent the holes that are not connected to the ground planes (NC).

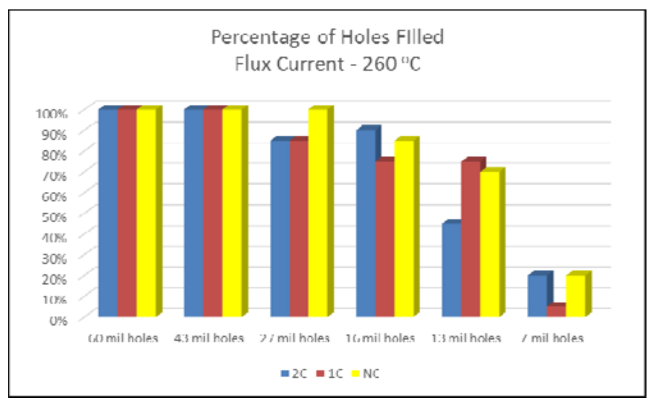

Figure 11: Hole fill results for the current flux at 260 °C

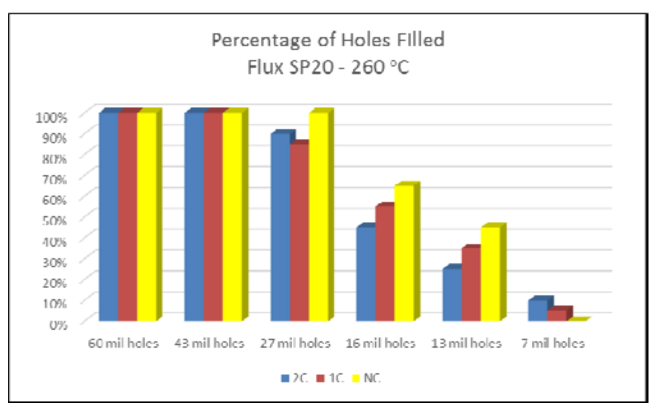

Figure 12: Hole fill results for the SP20% flux at 260 °C

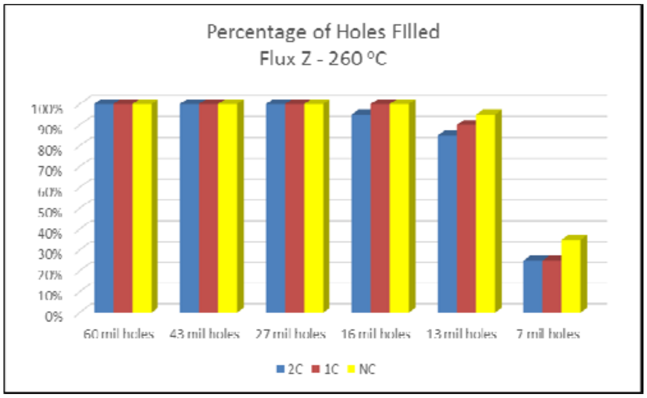

Figure 13: Hole fill results for flux Z at 260 °C

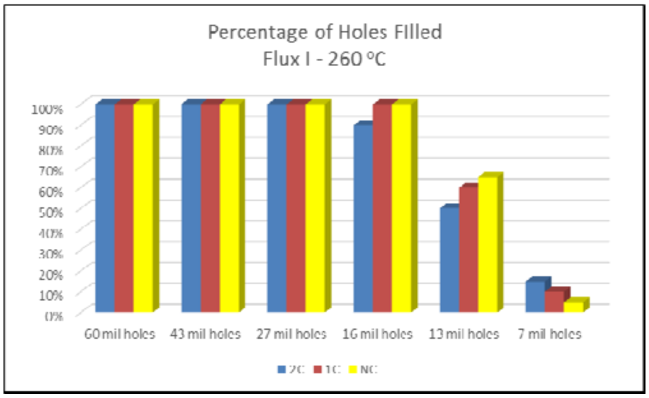

Figure 14: Hole fill results for flux I at 260 °C

Hole fill ratings of 100% were achieved by all fluxes for the 60 and 43 mil holes. The current and SP20% fluxes showed slightly worse performance for the 27 mil holes, while fluxes Z and I remained at 100%. Performance for the SP20% flux dropped off significantly for the 16 mil holes, while the other fluxes gave acceptable results. The 13 mil holes differentiated between the fluxes. Flux Z performed the best, followed by Flux I and the current flux, and the SP20% flux performed the worst. The 7 mil holes were too small to effectively wet with flux and solder. This caused the percentages of holes filled to be lower than 20% in most cases.

Another trend was observed with this test board design. The holes with two ground plane connections were more difficult to solder, followed by the holes with 1 ground plane connection. The holes without ground plane connections gave the highest fill ratings.

This test was repeated with a solder temperature of 280 °C. The results are in Figures 15 through 18.

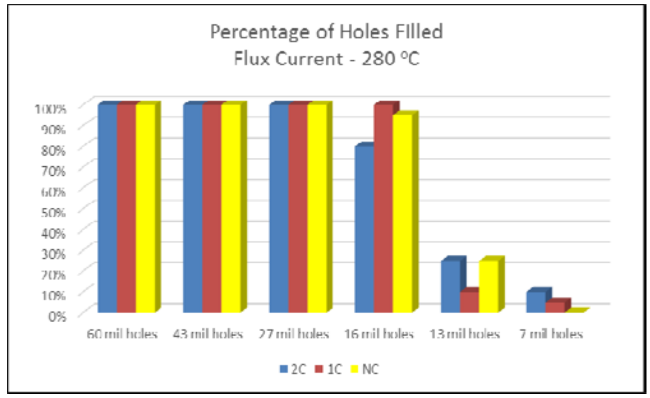

Figure 15: Hole fill results for the current flux at 280 °C

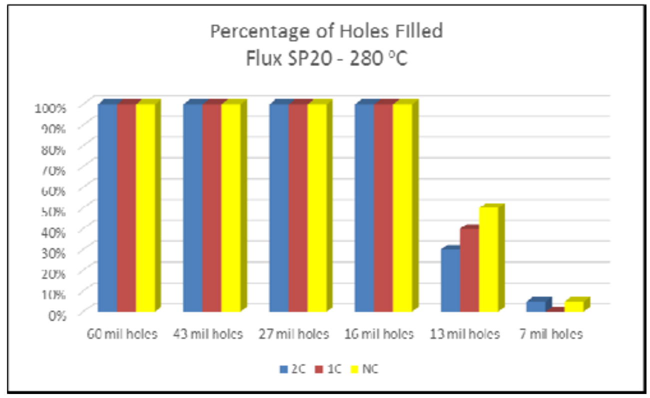

Figure 16: Hole fill results for the SP20% flux at 280 °C

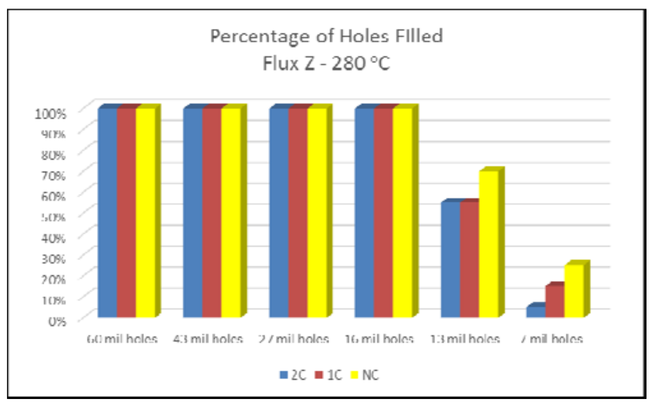

Figure 17: Hole fill results for flux Z at 280 °C

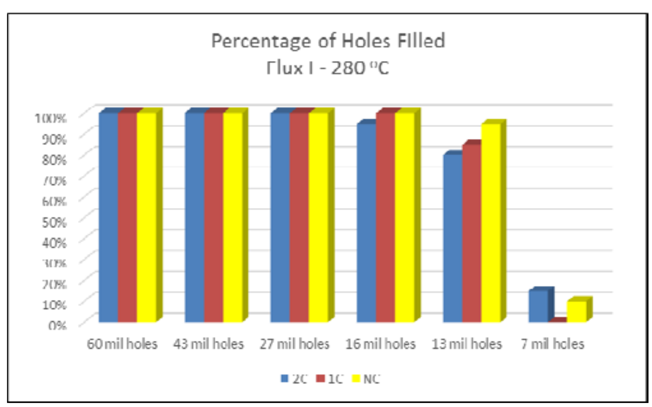

Figure 18: Hole fill results for flux I at 280 °C

Again the performance of all fluxes was excellent for the 60 mil, 43 mil, and 27 mil holes. The current flux did not perform as well as the others in the 16 mil hole size. The 13 mil hole size is again the main differentiator between the fluxes. Flux performance was the best with flux I, followed by flux Z, SP20% and the worst performance was shown by the current flux. The 7 mil holes were too small to show useful results.

The same basic trend of thermal demand on solder performance can be seen here. The holes with no ground connections soldered better than the holes with ground connections. This difference was less pronounced at 280 °C than at 260 °C, due to the fact that more heat is present in the solder.

This test was repeated with a solder temperature of 300 °C. The results are in Figures 19 through 22.

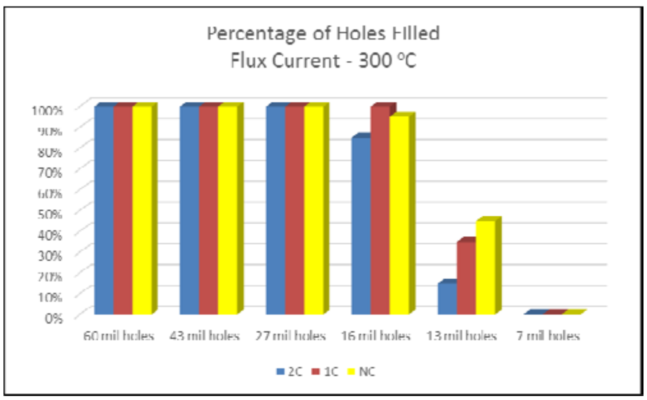

Figure 19: Hole fill results for the current flux at 300 °C

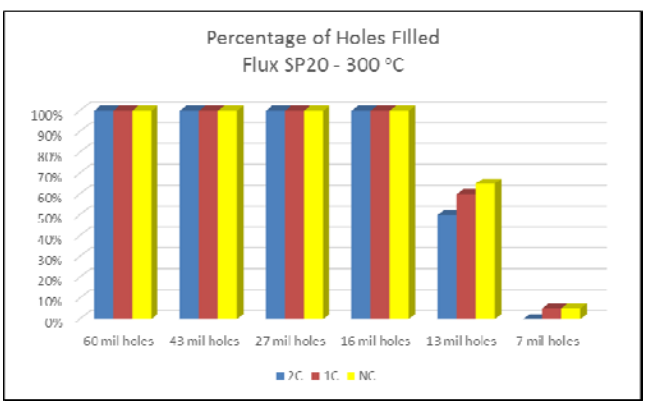

Figure 20: Hole fill results for the SP20% flux at 300 °C

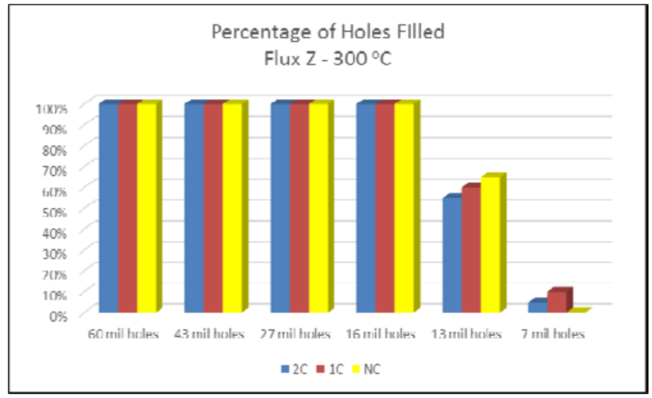

Figure 21: Hole fill results for flux Z at 300 °C

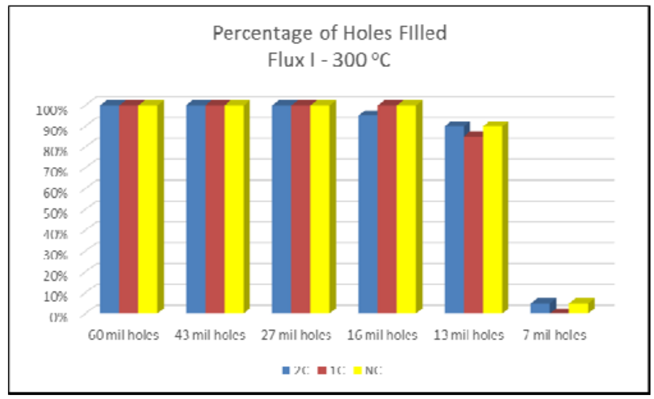

Figure 22: Hole fill results for flux I at 300 °C

The 60 mil, 43, mil, 27 mil, and 16 mil holes all received fill ratings of nearly 100% for all of the fluxes. The 7 mil holes again could not be effectively soldered in this test.

The 13 mil holes can be used to differentiate between the fluxes. Flux I performed the best with nearly 85% of the holes filled. Flux Z and SP20% gave similar performance with between 50 and 60% of the holes filled. The current flux had the worst performance at this temperature with only 10 to 40% of the holes being filled. The holes with two ground connections had lower hole fill ratings than the holes with one or no ground connections. This difference for the current flux was 35%, the SP20% flux was 15%, flux Z was 10%, and flux I was only 5%. This indicates that flux I is better able to wet and withstand the heat required to effectively solder the most thermally demanding holes.

Combining the data for the current flux for all three temperatures shows some interesting trends (Figure 23).

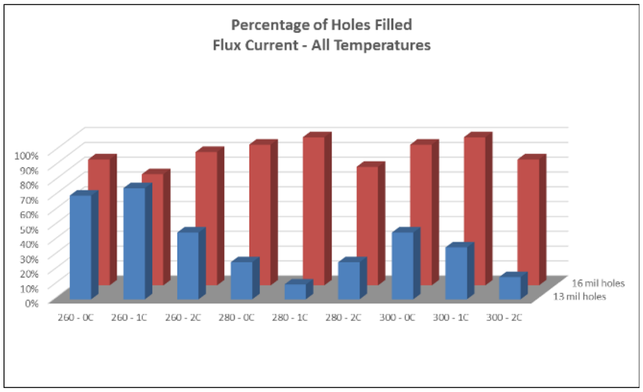

Figure 23: Hole fill results for the current flux at all temperatures.

The current flux solders 16 mil holes with good hole fill ratings. Increasing temperature increased the ratings slightly. This trend is quite different for the 13 mil hole size. Increasing temperature reduced the hole fill percentages dramatically. This same data combination for flux I shows a different trend (Figure 24).

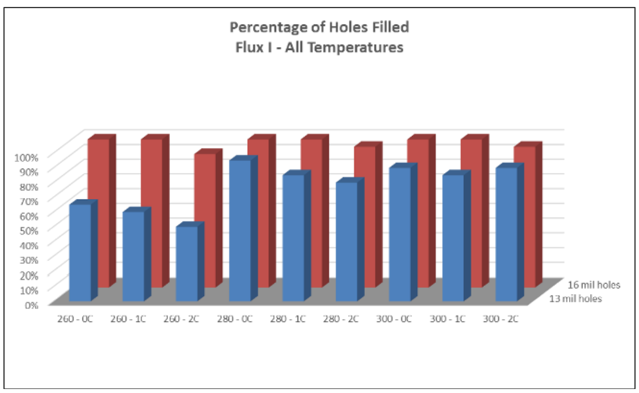

Figure 24: Hole fill results for flux I at all temperatures.

Hole fill ratings for flux I were higher overall than for the current flux. Increasing the temperature increased the hole fill percentages. Flux I is better able to wet smaller holes and is more thermally stable than the current flux.

Beta Site Test Results

Beta site testing was done with several of these flux formulations. Lead free selective and wave soldering was used along with very thermally demanding circuit boards. This testing lead to formulation refinements throughout the course of this project. Repeated laboratory and beta site testing resulted in one flux formulation being chosen for more widespread testing. The chosen flux formulation is being run in a longer term test in leaded and lead-free selective and wave soldering operations. Several additional beta sites have been included and testing is currently ongoing.

CONCLUSIONS

This process of formulation created a new water soluble liquid flux that meets the desired criteria. This flux works well with wave soldering temperatures of up to 300 °C and selective soldering temperatures of over 315 °C. Hole fill results are good even with high aspect ratio holes. The residues are easy to wash off with DI water and create minimal foam. The new flux is completely halide and halogen free, which is relatively new technology for water soluble liquid fluxes. Beta site testing has shown that this flux is usable in leaded and lead-free applications, and in wave and selective solder systems.

FUTURE WORK

Beta site testing is ongoing at a variety of sites. Multiple alloys are being used including Sn63/Pb37, SAC305, and SN100C®. Temperatures both above and below those used in this work will be tested. Feedback will be used to continue optimization of this new water soluble liquid flux.

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